Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

ACTIVITIES OF NATIONAL AND INTERNATIONAL STANDARDS ORGANIZATIONS RELEVANT TO CANADIAN COAL INTERESTS

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Prepared under the auspices of the American Society for Testing and Materials (ASTM) Committee D05 on Coal, Coke and Gaseous Fuels

ENERGY RESEARCH PROGRAM
ENERGY RESEARCH LABORATORIES
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MICROMEDIA

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FOREWORD

The market value of coal and the progress of research related to technologies employing coal as a substitute feedstock for conventional fuels are in part evaluated on the basis of analytical data obtained from procedures developed by national and international standards groups. In Canada, public and private sector organizations involved in the production, utilization, conversion and assessment of coal employ both American Society for Testing and Materials (ASTM) and International Standards Organization (ISO) methods to evaluate the physical and chemical properties of coal.

These procedures are continually reviewed and revised to respond to the need to assess wider ranges of coal quality or to evaluate properties which have an impact on novel technological applications. To provide a coordinating role for the maintenance and development of coal standards in Canada and to ensure that new and revised procedures implemented by national and international standards organizations are compatible with Canadian interests, the Energy Research Laboratories of CANMET participates actively in the deliberations of both ASTM and ISO. This participation includes advice and consultation on proposed revisions as well as the implementation of new test methods and specifications. In addition, candidate coals and laboratory services required to generate the experimental data upon which the progress of standards are based, are provided. This report is intended to provide a description of the structure, scope and current activities of ASTM Committee D05 on Coal and Coke. Subsequent reports will address the activities of ISO and the standards development process.

Since both ASTM and ISO procedures are subject to cyclic review, it is hoped that these reports will appear on a continuing basis so that the coal community may be kept aware ofthe most recent developments with respect to national and international coal standards.

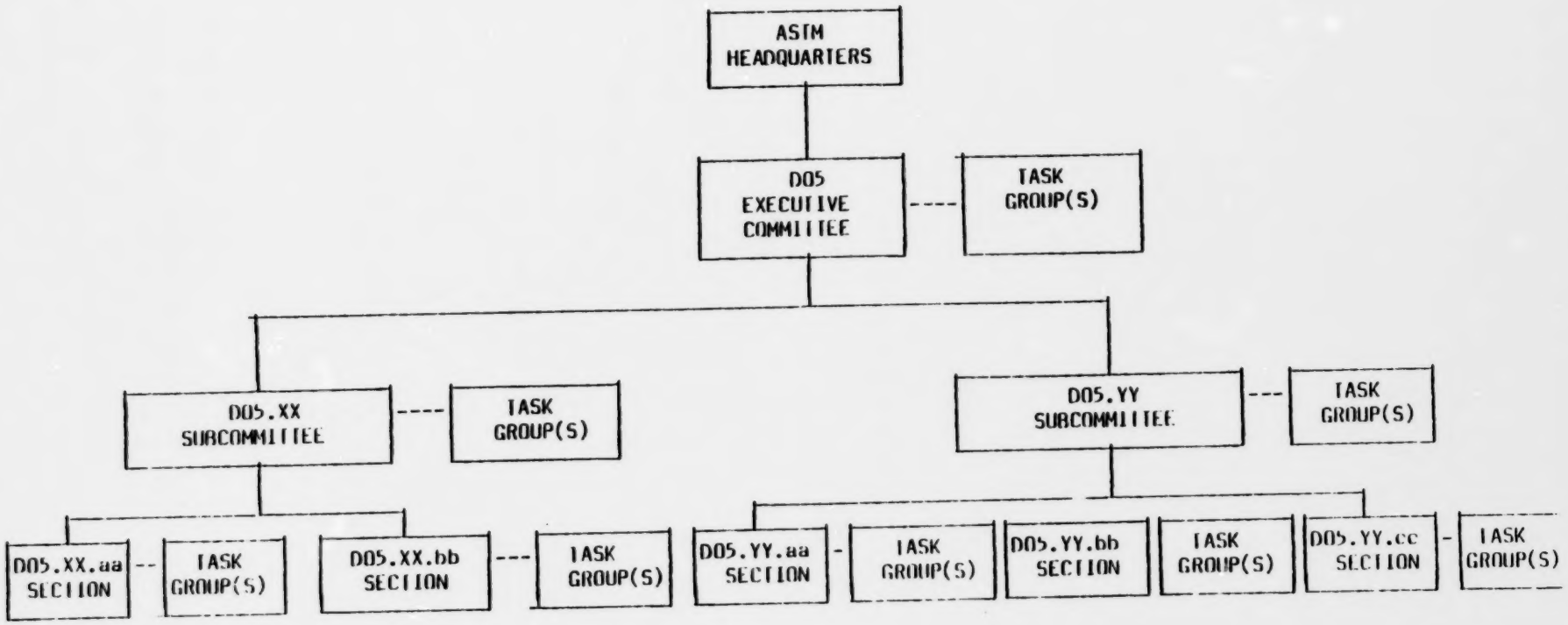
AMERICAN SOCIETY FOR TESTING
AND MATERIALS
COMMITTEE D05 ON COAL AND COKE

AN OVERVIEW AND STRUCTURE

ASTM is a non-profit corporation, which provides a technical forum and management system for the development of consensus standards on characteristics and performance of materials, products, systems, and services and the dissemination of related knowledge. The standardization and development work is done in technical committees which now number 139. Committee D05 on Coal and Coke is one such committee and currently has 315 members.

Committee D05 is responsible for the development of sampling, analysis and testing procedures for coal and coke from coal, as well as classification systems for coals based on specific physical and chemical properties. The committee is subdivided into subcommittees, sections and task groups organized as illustrated on page 2. These groups deal with standards related to terminology, sampling, physical and chemical analysis, classification, statistics, and accreditation as they apply to coal. Members of these subcommittees, related sections and their task groups, contribute on a voluntary basis whether representing their sponsoring organization or participating on an individual basis. In addition, members are classified as producer, user or general interest. Although the number of general interest

COMMITTEE STRUCTURE OF ASTM D05



1) All technical matters within the framework of D05 are assigned by the executive to the appropriate subcommittee, section or task group.

2) The technical subcommittees through their chairmen communicate with the executive concerning the activities of the subcommittee, the sections, the task groups and any other subordinate body working within the framework of that subcommittee.

3) Task groups may be set up by the executive committee, subcommittees or sections to investigate a particular technical aspect within the framework of the delegating body. However, task groups must be disbanded on completion of assigned duties.

members consisting of representatives of government, academia and service organizations is not limited ASTM regulations state that the number of producers may not exceed the combined total of users and general interest members to ensure the development and maintenance of objective standards. The scope of the subcommittees of Committee D05 and where applicable, the items delegated to their associated sections are listed on pages 7-20.

Within the framework of D05, there are two major ongoing activities:

(a) review and revision of existing standards,
(b) development of new standards.

These functions are coordinated and directed by the executive subcommittee. Work related to these two activities is assigned to the relevant subcommittee, which in turn may refer the issue to a section. If the appropriate group agrees that a revision or a proposal for a new standard warrants investigation, then a task group is formed to investigate the matter. It is within the task groups of Committee D05 where editorial and technical concerns are resolved. On pages 21-58 a summary of the task groups currently active within D05 is presented. For some task groups, information related to objective status and future directions is not available. However, this information will appear in the next report on ASTM activities. Because of their importance a brief description of the structure and some of the current issues facing ASTM task groups is provided below.

IMPORTANCE OF TASK GROUPS

Task groups are formed by subcommittees and sections to resolve matters related to the revision and development of standards which are of interest to the committee members. Because these members are representatives of organizations or individuals with interest in coal, the topics under investigation tend to be in harmony with those of concern to the coal community in general.

Task groups are generally formed to address:

(a) editorial revisions to existing standards,
(b) technical revisions to existing standards,
(c) development of new standards.

For cases (b) and (c), there is the additional requirement to support technical revisions and new standards with experimental data. From these data, procedural bias statements as well as precision statements concerning the range of application of the revised or new procedure and the variability in analytical results within and between laboratories are formulated. This requirement involves soliciting the participation of organizations having the facilities and personnel to carry out the experimental tests. In addition, candidate coals covering both the anticipated range of the parameter in question and the expected spectrum of coal rank must be selected. In all cases mentioned above, a research report, which is appended to the standard in question, is filed by the task group. To contribute to the activities of a task group, the participants do not need to be members of D05 unlike the requirement for subcommittees and sections. This provision, encourages D05 task groups to solicit contributions from

individuals and organizations which may have expertise or equipment which might help the task group attain its goal. When a task group has achieved its objective, it is dissolved.

There is a considerable concentration of effort within D05 to re-evaluate the precision statements attached to current standards and to incorporate instrumental techniques into the existing framework of D05 procedures. This is the result of several driving forces. First, a review of current ASTM D05 standards has indicated that not all existing precision statements were generated using consistent statistical criteria. This compounds the process of comparing related parameters of coal quality generated from different standards or even the same parameter generated from different standards. Second, since the mid-70's there has been a substantial increase in the utilization of lower rank coals as an alternate energy source, particularly for power generation purposes. Unfortunately, a significant number of the D05 standards did not include lower rank or low quality coals in the round robin programs designed to generate data for the original precision statements. To rectify this problem, the executive of D05 has directed subcommittees to reassess the precision statements for all standards within their jurisdiction according to well defined procedures approved by ASTM for conducting round robin programs. Where necessary, task groups are formed to produce a revised precision statement. This may occur at the time of cyclic review of the standard or if the opportunity arises, the standard may be reassessed in conjunction with a new alternative procedure.

Consideration of alternative procedures for incorporation into "classical" ASTM procedures or approval as entirely new standards has been promoted by the increase in the availability of state of the art microprocessor controlled instrumentation. These instrumental techniques often provide less labour intensive and more rapid methods for the determination of parameters of coal quality.

COMMITTEE D05 ON COAL AND COKE

SCOPE

The development of specifications for coal and coke produced from coal. The development of sampling, analysis, and testing procedures and the development of classifications of coals on the basis of chemical and physical characteristics. The standardization of terminology and the promotion of research and dissemination of knowledge in these fields.

The work of this committee shall be coordinated with other ASTM committees and other organizations having common interests.

EXECUTIVE OFFICERS

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SUBCOMMITTEES

SUBCOMMITTEE D05.02.00
NOMENCLATURE, DEFINITIONS AND EDITORIAL

CHAIRMAN

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SECRETARY

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SCOPE

To serve as editorial advisor to all technical subcommittees and to review the final draft of each proposed standard before balloting by Committee D05. To act as arbitrator and advisor in all matters pertaining to nomenclature and definitions appearing in the standards under the jurisdiction of this committee. To review all standard definitions for which this committee is responsible and make appropriate recommendations to this committee. To serve as direct liaison for the committee on editorial matters on all ASTM coal and coke standards with the Society's Standard Editor. Providing the Chairman and the Secretary of this committee with copies of all correspondence.

SECTIONS OF SUBCOMMITTEE D05.02.00

1. NONE

SUBCOMMITTEE D05.07.00
PHYSICAL CHARACTERISTICS OF COAL
AND BENEFICIATION OF COAL AND COAL SLURRIES

CHAIRMAN

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Tennessee University
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Tullahoma, TN 37388
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SECRETARY

Ronald Graham
Amax Coal Co.
5325 Oak Grove Road
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214-233-2911

SCOPE

To study and develop specifications and test methods for evaluating physical characteristics of coals, coal slurries and coke and to make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.07.00

1. D05.07.01 Beneficiation and Size Analysis
2. D05.07.02 Grindability and Physical Properties
3. D05.07.03 Slurry Characterization

SECTION D05.07.01
BENEFICIATION AND SIZE ANALYSIS

CHAIRMAN

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SECTION D05.07.02
GRINDABILITY AND PHYSICAL PROPERTIES

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SECTION D05.07.03
SLURRY CHARACTERIZATION

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SUBCOMMITTEE D05.15.00
PLASTICITY AND SWELLING OF COAL

CHAIRMAN

William Jasulaitis

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SCOPE

To study and develop test methods related to the plasticity, swelling and expansion characteristics of coals and to make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.15.00

1. None

SUBCOMMITTEE D05.18.00
CLASSIFICATION OF COALS

CHAIRMAN

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Ill State Geological
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Champaign, IL 61820

SECRETARY

SCOPE

To study and develop classifications of coals and to make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.18.00

1. None

SUBCOMMITTEE D05.21.00
METHODS OF ANALYSIS

CHAIRMAN

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SCOPE

To study and develop methods for the chemical analysis of coal, coal slurries and coke and to make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.21.00

1. D05.21.01 Proximate Analysis
2. D05.21.02 Ultimate Analysis
3. D05.21.03 Miscellaneous Analysis

SECTION D05.21.01
PROXIMATE ANALYSIS

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SECTION D05.21.02
ULTIMATE ANALYSIS

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SECRETARY

SECTION D05.21.03
MISCELLANEOUS ANALYSIS

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SUBCOMMITTEE D05.23.00
SAMPLING

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SECRETARY

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University
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SCOPE

To study and develop methods for sampling coal and coke, including methods for reducing any gross sample to the quantity and condition required for analysis or testing. To make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.23.00

1. D05.23.01 Coal Sampling
2. D05.23.02 Coke Sampling
3. D05.23.03 Sample Preparation

SECTION D05.23.01
COAL SAMPLING

CHAIRMAN

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SECTION D05.23.02
COKE SAMPLING

CHAIRMAN

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SECRETARY

SECTION D05.23.03
SAMPLE PREPARATION

CHAIRMAN

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SECRETARY

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SUBCOMMITTEE D05.24.00
STATISTICS

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SCOPE

To establish acceptable practices for obtaining and analyzing precision and bias test data related to coal and coke test methods. To assist in the other subcommittees of Committee D05 in establishing precision limits. To examine for validity the precision statements in proposed standard methods or revisions thereof related to coal coke and to make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.24.00

1. None

SUBCOMMITTEE D05.25.00
CHEMICAL AND PHYSICAL TESTING OF FUEL AGGLOMERATES DERIVED FROM COAL OR CHARCOAL (INACTIVE)

SCOPE

To study and standardize methods for evaluating physical and chemical properties of fuel agglomerates and to make appropriate recommendations to Committee D05.

SUBCOMMITTEE D05.27.00
USA ADVISORY COMMITTEE TO ISO/TC27 SOLID MINERAL FUELS

CHAIRMAN

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SECRETARY

Richard Muter
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SCOPE

To serve as the USA participant, through USA, in the activity of Technical Committee 27, Solid Mineral Fuels, of the International Standards Organization. In pursuing responsibility the duties of the subcommittee shall include:

1. Developing for ANSI recommended actions on various ISO/TC27 documents, including ISO Draft Proposals and ISO Draft Recommendations.

2. Recommending the USA delegates to meetings of ISO/TC27 and its subcommittees and working groups for approval by the D05 Executive Committee.

3. Developing and evaluating proposed international standards in cooperation with various laboratories.

4. Recommending the financial requirements for the support of ISO/TC27 participation to the Executive Committee of Committee D05.

SECTIONS OF SUBCOMMITTEE D05.27.00

1.	D05.27.02 Nomenclature, Definitions and Editorial	B. Stine
2.	D05.27.07 Physical Characteristics of Coal	C. Wagoner
3.	D05.27.15 Plasticity and Swelling of Coal	W. Jasulaitis
4.	D05.27.18 Classification of Coals	D. Mason
5.	D05.27.21 Methods of Analysis	J. Rose
6.	D05.27.22 Physical Tests of Coke	J. James
7.	D05.27.23 Sampling	G. Gould
8.	D05.27.24 Statistics	P. Bogardus
9.	D05.27.28 Petrographic Analysis of Coal	K. De Vanney
10.	D05.27.29 Major Elements in Ash and Trace Elements in Coal	J. Kuhn
11.	D05.27.30 Accreditation	F. Walker

SUBCOMMITTEE D05.28.00
PETROGRAPHIC ANALYSIS OF COAL

CHAIRMAN

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SECRETARY

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SCOPE

To study and develop specifications and test methods for determining the petrographic composition and related optical characteristics of coals and to make appropriate recommendations to Committee D05.

SECTIONS OF SUBCOMMITTEE D05.28.00

1. None

SUBCOMMITTEE D05.29.00
MAJOR ELEMENTS IN ASH AND TRACE ELEMENTS IN COAL

CHAIRMAN

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SECRETARY

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SCOPE

The standardization of methods for the determination of the major ash forming elements in coal, excluding sulfur and forms of sulfur. The determination of the major element composition of coal ash prepared under standardized conditions, and the determination of trace elements in coal or where complete retention has been proven in coal ash prepared under standardized conditions.

SECTIONS OF SUBCOMMITTEE D05.29.00

1. D05.29.01 Atomic Absorption Methods
2. D05.29.02 X-Ray Fluorescence Methods
3. D05.29.03 Other Methods

SECTION D05.29.01
MAINTENANCE OF EXISTING TEST METHODS

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SECTION D05.29.02
EVALUATION OF NEW TEST METHODS

CHAIRMAN

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SECTION D05.29.03
REFERENCE MATERIALS, CALIBRATION AND STANDARDIZATION

CHAIRMAN

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SUBCOMMITTEE D05.30.00
ACCREDITATION

CHAIRMAN

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SECRETARY

SCOPE

SECTIONS OF SUBCOMMITTEE D05.30.00

1. None

SUBCOMMITTEE D05.90.00
EXECUTIVE SUBCOMMITTEE

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SCOPE

SECTIONS OF SUBCOMMITTEE D05.90.00

1. None

Subcommittee 02.00 Nomenclature, Definitions and Editorial

No active task groups.

Subcommittee 07.00 Physical Characteristics of Coal and Beneficiation of Coal and Coal Slurries

No active task groups.

Section 07.01 Beneficiation and Size Analysis

1. Froth Flotation

Objective: To develop a new standard to establish the relative floatability of coal, e.g., fresh versus oxidized. Hopefully industry will be able to correlate the "relative floatability" with design elements of coal preparation plants or even obtain a measure of the quality of the product that would be obtained and the reagents necessary.

Status: Proposals upon which the standard will be based are being considered.

Future Directions: A proposed test method will be circulated to task group members for technical and editorial comment to be followed by a round robin program to obtain experimental data necessary to support the proposed standard.

2. Magnetite in Coal

Objective: To develop a standard test method for measuring the amount of magnetic magnetite in a sample of coal or magnetite using a Davis Tube Magnetics Tester.

Status: A draft standard is being formulated.

Future Directions: Editorial and technical review of the draft standard and round robin studies.

3. Size Analysis

Objective: Commercial producers and users of coal make use of a variety of ASTM D05 standards for selling and purchasing of coal by size (D 310, D 311, D 410, D 431). To avoid confusion and repetitiveness, the task group is attempting to produce one comprehensive standard which will elaborate more on wet versus dry screening. However, it is important to note that material below 500 mesh and pulverized coal for electricity generation purposes, will be excluded.

Status: A draft standard has been formulated, circulated and balloted.

Future Directions: Editiorial and technical review and round robin studies.

4. Washability:

Objective: Specifically to perform a round robin on 1/4" x 30 mesh material with a view to incorporating the results into the recently approved standard on "Washability" D 4371 which was first published in 1984.

Status: Organizations having facilities to carry out the necessary experimental work have been identified, and 4 coals have been distributed to 18 candidate organizations.

Future Directions: Two more candidate coals will be selected and distributed to complete the round robin work.

Section 07.02 Grindability and Physical Properties

1. Bulk Density by Nuclear Methods

Report not available.

2. Grindability Standard Sample Task Group

Objective: The calibration of grindability machines depends upon the proper testing of four suitably prepared standard reference coals. The purpose of the task group is to prepare quality assurance guidelines for both the authority responsible for the preparation of the standard reference samples and the user of the grindability standard.

Status: The task group has formulated a set of guidelines which are under review.

Future Directions: Publication of the guidelines in the near future.

3. Volumetric Determination of Coal in Storage

Report not available.

4. Determination of Tons of Coal in Storage

Report not available.

5. Sampling and Preparation of Samples for Determination of Grindability by D 409

Report not available.

Subcommittee 15.00 Plasticity and Swelling of Coal

1. Coal Oxidation

Report not available

2. F.S.I.

Report not available

3. Geisler Plastometer

Report not available

4. Sole Heated Oven

Objective: To develop a significance statement for standard D 2014 last reapproved in 1983. In addition, the task group is investigating the suitability of the precision statement attached to the current standards. If warranted, a round robin program will be undertaken to gather data to establish a new precision statement.

Status: A significance statement has been developed and a round robin is underway.

Future Directions: The round robin data will be employed to generate a new precision statement for the standard in time for its next reapproval in 1988.

5. Coke Reactivity

Objective: A variety of "industry" sponsored tests are employed to establish coke reactivity. Domestic and foreign buyers are employing these "non standard" tests for the purpose of selecting metallurgical coals to produce blast furnace coke. The primary objective of the task group is to develop a standard test method for the measurement of coke reactivity and after reaction strength properties of blast furnace coke.

Status: The task group is attempting to adopt a modification of the Nippon Steel method with improvements and certain elements present in current North American practices which will make the test more affordable while permitting a correlation of coke reactivity as made in accordance with the Nippon procedure and those made following the Bethlehem Steel technique. This approach will make the test more useful to North American users. At present coke reactivity and after reaction strength procedures employed by North American Laboratories known to perform these tests are being summarized to provide a basis for the development of a draft procedure. Also a suite of samples covering the range of coke reactivity necessary to obtain relevant experimental data are being gathered, prepared and distributed to the task group participants.

Future Directions: The draft procedure and samples will be distributed to the task group members for preliminary testing.

Subcommittee 18.00 Classification of Coals

1. Revision of D 388 Standard Classification of Coals by Rank and Modification of D 2796 Megascopic Description of Coals and Coal Seams and Microscopical Description and Analysis of Coal

   Objective: The degree of metamorphism of coal is expressed in terms of coal rank and has an impact on chemical and physical properties which are employed as estimates of behaviour in coal production and utilization. The task group is attempting to establish the effect of maceral content, in particular exinite and intertinite on classification parameters and to arrive at an acceptable definition of inertodetrinite (small pieces of inertinite) which presumably affects the cell walls of coke differently from larger particles. This would have a direct impact on the petrographic characterization of coal for coking purposes.

   Status: A definition of inertodetrinite has been formulated and will be balloted for acceptance by D05 members. Work on the effects of maceral content on classification parameters is ongoing.

   Future Direction: Addition of the definition of inerto detrinite to D 2796 and preparation of an ASTM D5 position paper on the need for research on the properties of macerals in North American Coals.

2. Investigations of Problems Imposed by the Effects of Mineral Matter on Coal Classification

Objective: Coals having a dry mineral matter free fixed carbon content of less than 69% are classified on the moist mineral matter free gross calorific value basis. In most cases, the mineral matter content is estimated from the ash content of the coal rather than determined experimentally. For impure coals (coals in excess of 25 wt% but less than 50 wt% ash) or for coals containing significant quantities of exchangeable cations, pyrite, carbonates, or expandible clays, the estimation of the mineral matter content according to accepted procedures can lead to misclassification.

Status: A questionnaire has been circulated to D05 members for comments on problems associated with estimation of mineral matter, proposed alternatives and interest in participating in work on the problems.

Future Direction: Formulation of proposals for estimation of parameters on the mineral-matter-free basis content, compilation of existing data, preparation of revisions to D388, and recommendations for experimental work if appropriate.

Subcommittee 21.00 Methods of Analysis

1. Methods for the Determination of Coal at 3/8" Top Size (also Task Group of 23.00)

Report not available.

Section 21.01 Proximate Analysis

1. 8-Mesh Tolerance Limits

Objective: To establish 8-mesh precision data for specified tests such as ash and heat value content. Except in the case of moisture (D 2961, D 3302) precision data are only available for the 60-mesh laboratory sample. These precision data which are a measure of the expected variability in analytical results, cannot be directly translated to the 8-mesh sample.

Status: A procedure for the parameters of interest has been issued and reviewed by the statistics subcommittee which has provided advice on the division and issuing of splits which should aid in the formulation of a proper precision statement.

Future Direction: Round robin program to gather data on 8-mesh coal needed to generate precision statements for the parameters of interest.

2. Revision of D 1412 Equilibrium Moisture

Report not available.

3. Shortening of Air Dry Moisture Test

Objective: To determine within a reasonable time frame whether a coal meets the minimum specifications required by the coal user prior to shipment, coal producers must have access to a rapid method for the determination of air drying loss. Current procedures, as specified in D 3302 and related standards (D 2961), are time consuming. The purpose of the task group is to establish a rapid air drying procedure which will have the additional benefit of minimizing the adverse effects of air drying time on readily oxidized coals.

Status: The task group has recommended a change to section 9.2.3 of D 3308.82 which indicates that thinner depths of coal in the drying pan can reduce drying times by as much as two thirds for some coals. The preparation of a method to air dry samples by using a microwave is currently underway.

Future Directions: Selection of coals and completion of a round robin program for the microwave technique.

4. Quantity Required for Moisture Determination
(also Task Group of 23.03)

Objective: The time required to carry out a total moisture determination for coal could be significantly reduced by minimizing sample reduction steps. This could be achieved by reducing the minimum amount of sample required at each stage of reduction or by increasing the minimum top size.

Status: Round robin work is being conducted on the minimum amount of sample required to arrive at a reliable moisture for 8-mesh coal.

Future Directions: Studies will be carried out on the effect of increasing minimum top size to 10 mm on the total moisture determination.

5. Modified Volatile Matter

Objective: The current ASTM method for the determination of volatile matter (D 3175) does not consistently produce the expected variability in results as indicated in the existing precision statement particularly for low-rank coals (lignites, subbituminous). The objective of the task group is to develop a procedure which will produce results of consistent variability.

Status: An initial round robin program was conducted employing a two-stage furnace procedure. Results of the round robin suggested that alternate procedures might be required for certain coals and that crucibles fabricated from different materials and of different construction gave variable results.

Future Directions: The task group has decided to use only platinum crucibles in round robin studies and is currently re-evaluating the proposed procedure.

6. Vapourization of Coal by TGA

Objective: The rate of loss of volatile components of coal when heated in an inert atmosphere or the susceptibility of coal to ignition in an oxidizing atmosphere might be useful in predicting the suitability of coal for various combustion and conversion processes. The purpose of the task group is to determine whether current thermogravimetric analytical techniques can produce experimental information which might be useful predictors of the combustion or conversion properties of coal.

Status: The task group was recently formed and is seeking participants having the necessary instrumentation and expertise required to produce acceptable results.

Future Directions: Selection of parameters to be measured and formulation of a proposed procedure.

7. Proximate Analysis by Instrumental Methods

Objective: The increase in the availability of state of the art microprocessor controlled instrumentation, which provides a less labour intensive route for the determination of the moisture ash and volatile content of coal, has resulted in a reduction in the use of classical ASTM procedures for the proximate analysis of coal. The objective of the task group is to attempt to develop a procedure which will encompass all current instrumental techniques. As well, during the course of the round robin program required to provide experimental data in support of the new procedure, data will be gathered to assess the precision statements attached to the current procedures (D 3173-73, D 3174-82, D 3175-82).

Status: A procedure is being drafted for editorial and technical review by the task group participants.

Future Directions: Circulation of procedures and samples for a round robin study.

8. Type of Container for Determination of Moisture in the Analysis Sample

Objective: Data have been supplied by members of D05 indicating that a potential bias exists between moisture determined in glass versus porcelain crucibles. The objective of the task group is to investigate the causes and remedies for the alleged bias.

Status: The task group will solicit interest for participation.

Future Directions: Reassessment of current procedures.

Section 21.02 Ultimate Analysis

1. Instrumental CHN

Objective: The proliferation of instruments which have the capability to provide rapid analysis of carbon, hydrogen and nitrogen in solid fuel samples has resulted in a significant decrease in the use of current ASTM standards for the determination of these elements (D 3178, D 3179). In addition the precision statement attached to D 3178 for carbon and hydrogen does not address the expected variability for results between laboratories (reproducibility). The objective of the task group is to develop an instrumental ASTM standard.

Status: The task group has developed a proposed instrumental procedure. Candidate coals for a round robin study have been selected and will be circulated with an NBS reference coal of known composition which will be used for calibration.

Future Directions: The round robin program will produce data for generation of a precision statement including components related to repeatability, reproducibility, and bias.

2. Instrumental S

Objective: Similar to the situation for carbon, hydrogen and nitrogen, the availability of state of the art instrumental techniques had resulted in a drastic reduction in the use of the classical ASTM sulphur standard D 3177. The task group was requested to formulate a comprehensive procedure for the determination of S in coal by instrumental techniques.

Status: The task group successfully achieved its objective with the publication of D 4239-83. Final resolution of the wording of a note in standard should result in dissolution of the task group in the near future.

Future Directions: The work of the task group is complete.

3. Direct Oxygen:

Objective: No approved ASTM method exists for the direct determination of oxygen in coal. Oxygen is determined by difference after determining specified components by employing current proximate and ultimate methods. Problems concerning the changes in mineral matter components on ashing and the difficulty in establishing true organic nitrogen, carbon, hydrogen and sulphur contents result in significant errors in estimating the oxygen content in certain cases. Although classical procedures and sophisticated instrumental techniques exist for the direct determination of oxygen, they are in general extremely time consuming, and plagued by the difficulty in differentiating between organic and mineral oxygen. In addition, instrumental techniques generally involve the use of capital intensive apparatus. The objective of the task group is to develop a procedure which overcomes these problems.

Status: The task group has developed a procedure although participation in the work has been limited.

Future Direction: The intent is to place the method in the back of the 1986 Book of Standards, as a proposed standard.

Section 21.03 Miscellaneous Analysis

1. Forms of Sulphur

Objective: There is some question as to whether the procedure for pyritic sulphur in coal as specified in D 2492-84 results in the complete extraction of pyritic iron from certain coals. This is thought to be a result of the fact that in some coals the pyrite is finely disseminated in the organic matrix which prevents penetration of the acid used in the extraction. The task group is to investigate an alternative ashing procedure which would eliminate the "organic effect" and also to assess the precision statement attached to D 2492.

Status: Results on eight samples covering the entire spectrum of coal rank have been received from nine laboratories using both the alternative and existing procedure (D 2492). Highly variable within-laboratory results on one of the samples indicated segregation problems. Comments were also made on the lack of samples covering the middle range of anticipated pyritic iron concentration. As a result, task group members have been asked to undertake a second experimental phase involving the analysis of two additional samples covering the concentration "gap" and retesting of the sample which presented segregation problems. To date, results have been received from eight laboratories on the second phase.

Future Directions: Generation of a precision statement for the alternative procedure and reassessment of the precision statement for D 2492 on the basis of experimental data.

2. Definition of Isoperibol Calorimetry

Report not available.

3. Investigation of Existing and Novel Calorimetric Techniques

Report not available.

Subcommittee 23.00 Sampling

1. Methods for the Determination of Coal at 3/8" Top Size (also Task Group of 21.00)

2. Test Method for Bias

Objective: There has been a need for a standard test method for bias (systematic) error with relation to the procedures described in D 2234 "Collection of a Gross Sample of Coal" since the standard was adopted. The need has become more urgent as manufacturers of equipment, regulatory agencies and producers and consumers of coal have become more demanding of coal sampling. In the past, the tendency to suspend critical judgement when mechanical sampling was involved has given way to the recognition that performance of mechanical systems, particularly when large quantities of coal are involved, can be a source of consequential error and cannot be assumed to be free of bias. However, testing for bias involves more advanced statistical techniques than encountered in other applications. The need to produce a technically rigorous statistical procedure is compounded by the need for simplicity and tutorial support for the non-statisticians who must be able to employ a consensus standard in the real world. The purpose of the task group is to develop a standard test method for bias which incorporates these elements.

Status: The standard is presently a published D05 proposal D 109. As such, the proposal must be balloted for inclusion or withdrawal as an official D05 standard within two years of its approval. This was carried out in the spring of 1985 and two negative votes were received.

Future Directions: Efforts will be made to resolve the negative votes. If this is successful, the proposal may become an approved D05 standard.

3. Test Method Measuring Sampling and Sample Preparation Precision

Objective: To investigate and develop improved means of determining the sampling precision of mechanical systems. No procedures are described in ASTM standards for determining the precision or random error associated with mechanical systems that are generally accepted in the industry. The task group will attempt to develop a method based on the proper application of statistical principles that would be included in future standards. Such a method is needed by industry in order to assess the effectiveness of sampling systems and to optimize sampling efficiency.

Status: A method has been drafted and field data have been collected that indicate the method is feasible.

Future Directions: Editorial and technical review of the method and a redraft if necessary prior to balloting.

Section 23.01 Coal Sampling

1. Core Sampling

Objective: The task group intends to produce a standard that would address coal sampling in terms of drill core sampling for exploration and mine planning purposes. The completed standard would provide guidelines for producers and users to properly sample and generate useful and consistent data.

Status: A third draft of a special technical publication concerning drill coring is in progress.

Future Directions: From a final draft of the drill core publication, a core sampling standard will be extracted and condensed.

2. Full Stream Mechanical Sampling (also Task Group of 23.03)

Objective: No approved ASTM D05 standard exists that applies specifically to "on stream" sampling and sample preparation for mechanical systems. Users must refer to D 2234 "Collection of a Gross Sample of Coal" and D 2013 "Preparing Coal Samples for Analysis" for sampler design and operation. These standards were developed for manual sample preparation and are insuitable for mechanical systems. The purpose of the task group is to produce a standard which will eliminate

the need to employ D 2234 and D 2013 while providing for a reference to D 2013 for manual "off" stream sample preparation of the final mechanical sample.

Status: A procedure is being developed for discussion of format and content.

Future Directions: Review of procedure by task group members and if necessary round robin studies.

3. Manual Stationary Sampling

Objective: A large percentage of coal is sampled with stationary methods. Existing ASTM standard D 2234 "Collection of a Gross Sample of Coal" does not give specific details in describing procedures and equipment to be used for rail car, truck, barge and stockpiled coal. The purpose of the task group is to develop a separate standard for the above categories.

Status: A standard for manual stationary sampling from rail cars was developed. A draft standard was circulated for approval by subcommittee 23.00 in early 1985.

Future Directions: Valid technical and editorial comments received as a result of the ballot will be included in the proposed standard prior to submission to the D05 main committee for approval.

4. Mechanical Auger Sampling

Objective: There is an increasingly wide use of auger samplers in industry. These auger samplers fill a need that cannot be met with other types except at great expense in facility modification and operation. Existing standards do not adequately cover auger sampling and methods of testing for bias and precision would require somewhat different approaches and procedures than other sampling methods. The objective of the task group is to develop a standard specific to auger sampling systems.

Status: A number of negative votes were received on a draft of a proposed standard balloted within subcommittee 23.00 in January 1985. Suggested changes with merit are to be included in a new draft which will be reballoted. Negative ballots proposing that an auger standard not be developed at this time will be resolved within the Coal Sampling Section 23.01.

Future Directions: A decision on the most reasonable route for the development and approval of the standard on auger sampling.

5. Revision of D 2013 "Preparing Coal Samples for Analysis"and D 2234 "Collection of a Gross Sample of Coal" (also Task Group of 23.03

Objective: To revise the two standards to eliminate sources of controversy arising from potentially "confusing" language in the standards.

Status: A draft is being developed for balloting. Basic changes to the standards are being worked on by other task groups.

Future Directions: Reconciliation of ambiguous statements within the standards as a result of balloting.

6. Generic Standard Principles of Sampling and Sample Preparation (also Task Group of 23.03)

Objective: Much of the existing language in D 2234 "Collection of a Gross Sample of Coal" and D 2013 "Preparing Coal Samples for Analysis" originated many years ago based on developed data and principles and has been repeated in successive standards. The purpose of the task group is to develop a comprehensive generic standard on "General Principles of Sample Preparation" which would incorporate current practices as well as relevant concepts from the existing standards.

Status: A first draft is being developed for format and content discussion.

Future Directions: Editorial and technical review of the draft by the task group prior to balloting.

7. Minimum Sample Increment

Objective: There is a need for intermediate minimum increment mass between the existing 2" and 6" top size increments specified and there are no requirements in ASTM standards for minimum increment masses below 5/8". Specifying minimum increment masses for the full range of coal sizes in use would provide guidance that is not available in current ASTM sampling standards and would result in a uniform basis for design of manual and mechanical sampling systems. The purpose of the task group is to arrive at a consensus on minimum sample increments to assist in the development of the language for the proposed generic sampling standard.

Status: Progress has been made in developing compromise language and quantities that would satisfy the different views.

Future Directions: Selection of the range of increment masses for inclusion in the generic sampling standard.

8. Crusher Study (also Task Group of 23.03)

Objective: The moisture loss in sample crushing is an issue of great interest to operators of sampling systems. The purpose of the task group is to:

(a) Determine if sample crushers in use can produce a consistent 8-mesh product and whether modifications can be made to improve their performance in this respect.

(b) Investigate moisture loss in sample crushers for the range of conditions, sizes produced, and coal characteristics.

Status: It has been concluded that crushers cannot produce a consistent nominal 8-mesh product for most coals and crusher manufacturers have expressed the opinion that no modifications can be made to achieve the nominal 8-mesh product for most coals.

Future Directions: Special moisture test data should be developed which will help to evaluate moisture loss through crushers.

9. Part Stream Sampling (also Task Group of 23.03)

Objective: Sizeable quantities of coal are sampled with part stream methods. ASTM standard D 2234 "Collection of a Gross Sample of Coal" provides no details describing part stream procedures and equipment. The purpose of the task group is to develop special purpose part stream sampling procedures and equipment.

Status: Chairman has not been selected.

Future Directions: The task group will develop a draft for both manual and mechanical procedures.

Section 23.02 Coke Sampling

No active task groups.

Section 23.03 Sample Preparation

1. Generic Standard Principles of Sampling and Sample Preparation (also Task Group of 23.01)

2. Full Stream Mechanical Sampling (also Task Group of 23.01)

3. 8-Mesh Tolerance (also Task Group of 21.01)

4. Quantity Required for Moisture Determination (also Task Group of 21.01)

5. Revision of D 2013 and D 2234 (also Task Group of 23.01)

6. Crusher Study (also Task Group of 23.01)

7. Part Stream Sampling (also Task Group of 23.01)

8. Sample Dividers
   (report not available)

9. Plus 60-mesh Coal in the Laboratory Sample
   (report not available)

Subcommittee 24.00 Statistics

1. Protocol for the Generation of a Precision Statement

Objective: Precision statements attached to ASTM D05 standards are generated from experimental data collected by task groups investigating technical revisions to existing standards or developing proposed standards. These precision statements define the variability expected within (repeatability) and between (reproducibility) laboratories. It is important that these precision statements are generated in an efficient and consistent format. The objective of the task group is to produce guidelines defining the steps and statistical criteria all task groups should follow from the point of inception of a revision to a standard or a new proposal to generation of a precision statement.

Status: The task group has reviewed the ASTM statistical procedures recommended by subcommittee E on statistical methods. The task group has recommended that ASTM E-691 or procedures derived from E-691 (standard practice for conducting an interlaboratory test program to determine the precision of test methods) be used as a basis for the generation of precision statements for all D05 standards. The task group is attempting to extract and condense from those procedures, a document which will provide guidelines for the generation of a precision statement for all D05 standards.

Subcommittee 28.00 Petrographic Analysis of Coal

1. Coal Petrographic Training/Exchange Pellet Kits

Objective: Coal petrography methodology as employed in most laboratories includes a wide variety of techniques, equipment, and applications. The prime purpose of the task group is to distribute petrographic pellet training/exchange kits which would serve as an aid to training new laboratories and provide a forum for informational exchange between established laboratories conducting coal petrography. The program would also supplement the development of terms and methodology related to ASTM consensus standards for coal petrography applications.

Status: The task group has developed pellet kits which include four polished briquettes (two photomicrographs for each pellet with macerals labelled, as well as reflectance and macerals analysis. To date 25 kits have been distributed to public and private sector organizations throughout North America.

Future Directions: A survey of all organizations participating in the program to determine equipment and techniques employed for macerals analysis will be conducted by 1986. This information will be employed along with data supplied by the various participants to develop improved precision statements for existing ASTM methods.

2. Automated Petrography

Objective: The objective of this task group is to develop a standard for automated coal petrography.

Present Status: After reviewing the responses to a questionnaire on automated petrography, a task group was formed to continue work in this area. The group decided to issue the questionnaire on automated petrography to the Society of Organic Petrographers (SOP). The group will also correspond with The International Committee for Coal Petrography to monitor developments in Europe.

Future Directions: At the next meeting, the group will discuss the results of the questionnaire given to the SOP and an initial round robin using the ASTM pellet kits.

3. Coke Carbon Forms (Coke Petrography)

Objective: Coke petrography can be broadly subdivided into two categories: coke microtexture (or carbon forms) and coke microstructure (coke wall, pore associations). Various classification schemes for coke microtexture have been proposed and used internationally for years. In North America, there are at least three systems in the literature (US Steel, Bethlehem Steel and Inland Steel). The first objective of this task group is to develop a coke microtexture (carbon forms) classification and associated methods for ASTM. Subsequent objectives will include coke microstructural systems and methods.

Status: This task group was formed at the Fall 1984 ASTM meeting. Work was begun at the (Denver) Spring 1985 meeting whereby presentations by US Steel and Bethlehem Steel were given to the task group members and visitors. An explanation of the different coke carbon form systems was the main focus of the meeting with discussions on the applications of this type of analysis such as post mortem coal blend determinations, coke plant operating problems, coke reactivity implications as well as the relationship of coal rank and type to coke carbon forms. In August, an interim task meeting was held at US Steels Technical Center (Monroeville, PA). Assignments were made to begin an ASTM classification scheme. A co-chairman was also appointed. Task group members will be completing a form to identify the carbon forms grouping coupled with the various task group

members' interpretation of the morphological, optical, and physical properties. This will be available for task group review by the Spring 1986 meeting.

Future Directions: In the short term the group intends to have a tentative classification of coke carbon forms for blast furnace coke within one year. A coke round robin will then be initiated and procedures to prepare samples and conduct the analysis, etc. will be formulated. In the long term methodology for coke carbon forms as applied to foundry coke will be addressed as well as coke microstructure for ASTM standards.

Subcommittee 29.00 Major Elements in Ash and Trace Elements in Coal

No active task groups.

Section 29.01 Maintenance of Existing Test Methods

1. Cd, Ni, Pb Improvement of Sensitivity

Objective: The current ASTM D05 standard for the determination of nickel and lead D 3683-78 "Trace Elements in Coal and Coke Ash by Atomic Absorption" requires an initial ignition step which presumably results in a quantitative retention of these elements in the ash. No approved D05 procedure exists for the determination of cadmium. Application of techniques which would provide improved methods for the detection of these elements could permit a reduction in the lower quantitative limit for both nickel and lead compared with the current limits specified in D 3683 or perhaps permit their determination in coal without the need for a preliminary ashing step. The latter would reduce the potential for sample contamination as well as minimize concerns with respect to trace element volatility. Determination of these elements in coal would also assist in producing a comprehensive set of procedures for their estimation across fuel, mineral and atmospheric components when coal is used for power generation purposes or for evaluating the fate of these elements in conversion processes. The purpose of the task group is to assess techniques to achieve some of the goals outlined above.

Status: The task group is reviewing techniques applied to materials related to coal which might provide a significant improvement over the approved techniques for the detection of these elements.

Future Directions: Determination of the number of participants having the equipment and resources available to apply improved techniques to the determination of nickel, lead and cadmium in coal and coke. Preparation, technical and editorial review of the proposed procedure.

2. Substitution of HNO_3 for HCl in D 3682-78 "Major and Minor Elements in Coal and Coke Ash by Atomic Absorption"

Objective: It is recommended that the determination of phosphorus in coal and coke ash be carried out as described in D 2795-84 "Analysis of Coal and Coke Ash". Substitution of nitric acid for hydrochloric acid in D 3682-78 would facilitate the determination of phosphorus in the sample solution prepared according to D 3682 as this would minimize an interference created by the presence of iron. Furthermore, collection of comparative data on preparation of the sample solution by employing hydrochloric versus nitric acid would permit the reassessment of the precision statement attached to D 3682. The purpose of the task group is to collect data for the determination of the major and minor elements specified in D 3682 using both hydrochloric acid and nitric acid for preparation of the final solution and to use the data to address the issues described above.

Status: Preliminary comparative data have been submitted to indicate that the results obtained employing nitric acid are comparable to those obtained using hydrochloric acid.

Future Directions: The need to set up a rigorous round robin program to provide data for generation of an acceptable precision statement is to be discussed.

3. Examination of the Precision Statement of D 4326-84 "Major and Minor Elements in Coal and Coke Ash by X-ray Fluorescence"

Objective: To reassess the precision statement attached to D 4326-84 with a view to improving the precision for elements such as sodium and magnesium. To incorporate a precision statement for sulphur and to specify the analytical range for each element included in the procedure.

Status: Coal ashes covering the analytical ranges anticipated for North American coals for each element within the scope of D 4326-84 have been distributed for analysis.

Future Directions: Evaluation of the data according to criteria specified in E-691 to produce a new precision statement for comparison with the existing precision statement.

Section 29.02 Evaluation of New Test Methods

1. Raw Coal XRF

Objective: A procedure is needed which would permit the rapid determination of major and minor elements in coal without the need for destruction of the organic coal matrix. X-ray fluorescence offers promise as a non-destructive technique for the determination of major and minor elements in coals with the additional benefit of determining sulphur and chlorine which are lost during the time-consuming ashing steps prescribed in D 3682-78 "Major and Minor Elements in Coal and Coke Ash by Atomic Absorption", D 4326-84 "Major and Minor Elements in Coal and Coke Ash by X-ray Fluorescence" and D 2795-84 "Analysis of Coal and Coke Ash". The purpose of the task group is to develop an XRF procedure which could be applied to raw coal.

Status: A procedure has been prepared and a round robin program has been conducted. Experimental data from the program have indicated that the highly variable mineral and moisture contents of coal confound the process of assessing the results.

Future Directions: Corrections inherent to X-ray fluorescence techniques will be applied consistently to the results from various laboratories to arrive at a preliminary precision statement to be discussed by the task group.

2. Inductively coupled plasma (ICP)

Objective: Plasma emission spectrometry offers a rapid technique for the determination of major and minor elements in coal and coke ash. Apparently this technique is not plagued by the interference and matrix problems associated with alternatives such as atomic absorption and X-ray fluorescence. The purpose of the task group is to develop a procedure for the determination of major and minor elements in ash using ICP emission spectrometry.

Status: Two ashes were distributed to participants for analyses according to their in-house procedures. Results for elements were reported along with preparative techniques employed, and instrument parameter settings.

Future Directions: A comprehensive draft procedure including preparative techniques which appear applicable to ash from coal or coke and which specifies appropriate instrument parameters will be distributed for review by participants prior to a formal round robin program.

3. Volatile Sodium

Objective: From the point of view of power generation sodium in coal contributes significantly to boiler fouling if present in significant quantities. Sodium in coal is estimated by determining the sodium in the ash derived from the coal and then calculating the result back to the raw coal. There is some dispute as to whether certain sodium compounds which might be present in coal are volatilized during the ashing step resulting in a non quantitative retention of sodium in the ash and subsequently an incorrect estimation of the sodium in the raw coal. The purpose of the task group is to resolve this issue.

Status: A review of studies conducted on this issue is being carried out.

Future Directions: The task group will employ the results of the review to select coals for a round robin study if deemed necessary.

4. Ion Chromatography

Objective: Methods for the determination of chlorine (D 4208, D 2361), fluorine (D 3761) and sulphur (D 3177, D 2492, D 4329) have limited lower quantitative ranges thereby eliminating their application to coals containing elements below these ranges. No approved D05 method exists for the determination of phosphorus in coal or coke. Ion chromatography is an analytical tool which could significantly reduce the lower quantitative limits for some or all of the elements mentioned as well as providing a technique for the determination of phosphorus in coal and coke. The purpose of the task group is to develop an ion chromatography procedure to determine these elements in coal and coke.

Status: A procedure has been circulated for review by the participants. Coals are being selected for a round robin program.

Future Directions: Finalization of a proposed standard round robin program.

5. Sulphur in Ash

Objective: D 1757-80 is the approved method for the determination of sulphur in ash. The method incorporates three alternative procedures which are tedious and time consuming. The recent success in developing an instrumental procedure for the determination of sulphur in coal and coke employing high temperature combustion techniques (D 4239-83) has promoted interest in establishing a rapid instrumental technique for coal and coke ash. The purpose of the task group is to develop a procedure for coal and coke ash based on D 4239-83.

Status: Chairman has not been selected.

Future Directions: Selection of a chairman and development of a proposed procedure based on D 4239-83.

Section 29.03 Reference Materials, Calibration and Standardization

No active task groups.

Subcommittee 30.00 Accreditation

1. Guideline for Selection of an Evaluator

   Report not available.

Subcommittee 90.00 Executive

1. ASTM/EPA Joint Task Group on Quality Assurance for Analysis

   Report not available.

2. ASTM/EPA Joint Task Group on Quality Assurance for Sampling

   Report not available.

3. Peat as a Fuel

   Report not available.